Characterization of Sintered Metal Additive Manufactured Products

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Abstract— This research focuses on the application of 3D stereolithography (SLA) printing technology to metal printing, with copper powder serving as the primary material for the fabrication of intricate copper-based components. Copper is well known for its outstanding thermal and electrical conductivities, offering unique advantages across various engineering domains. Anycubic acrylate-based photopolymer resin and fine copper powder is used. The composition of the Cu-Resin-Glycol mixture was 52% copper powder, 28.8% resin, and 19.2% glycol. This composition was selected to achieve the highest possible density in the sample. Ethylene glycol was used as a diluent. It is observed that ethylene glycol is usable as it effectively reduces the viscosity of resin without altering the chemical nature and curing time of photocurable resin. By equating the viscosity of optimized Cu-resin mixture with pure resin, difference in curing times is calculated for finite thickness of layer. This shows that 28% increase in curing time is observed when Cu-resin mixture is used as compared to pure resin. A two-phase pressure-based sintering process is used in which controlled pressure is generated to reduce the proportion of voids and increase the density and structural integrity of the printed copper components, thereby ensuring the best possible material consolidation. Thus, the focus is to optimize various 3D printing parameters such as layer-by-layer thickness, exposure time, and curing techniques. [1] A two-step sintering process was also used to improve the density and strength of the material. Afterwards, characterization techniques, such as microstructural analysis and measurements of the thermal and electrical conductivities of copper, were carried out to analyze their different characteristics, including microstructure, grain size, and distribution.

These methods were used in addition to conventional testing to evaluate the structural integrity and mechanical behavior of copper samples, such as hardness, tensile strength, and ductility. This research study highlights the advantages of 3D-printed copper components over those manufactured using traditional methods. The results of this research hold significant potential for advancing 3D SLA printing technology and aim to foster progress in industries such as electronics, telecommunications, and energy systems that demand high-quality copper components with enhanced thermal and electrical conductivities.

Keywords: *3D SLA (stereo lithography) Printing, Copper, Sintering, Microstructure Analysis, Electrical and Thermal Performance Evaluation, Mechanical Testing*

Specialized Tools: *3D SLA Printer, Sintering Furnace, Testing Equipment*

I. INTRODUCTION

The ability to produce complex components using additive manufacturing (AM) technologies has revolutionized the manufacturing industry by reducing the limitations imposed by conventional methods of material removal. One of these cutting-edge techniques for producing metal stands out: 3D stereolithography (SLA) printing. This study focuses on using SLA technology to create copper-based components using copper powder as the main material. Due to its outstanding thermal and electrical conductivities, copper is extremely valuable in many technical domains. The main objective is to improve pure copper 3D metal printing and achieve the thickest layers possible using a two-stage pressure- based sintering procedure. [2] Applying regulated pressure during sintering can reduce the number of voids in printed copper components, improving material consolidation and structural soundness.

This research examines the mechanical characteristics and fatigue behavior of sintered copper printed items to offer a thorough assessment of their caliber. Key characteristics including hardness, tensile strength, and ductility are examined using standardized testing techniques, and the outcomes are compared to those of typically made items. Fatigue crack growth studies and cyclic loading tests are also used to look at the fatigue performance of sintered copper components. Examining the size, distribution, and general microstructure of the material is feasible using the microstructural analysis techniques of scanning electron microscopy (SEM).

In general, this research helps to advance SLA metal printing and other additive manufacturing techniques. This study prepares for improved usage of copper in many projects, advancing development and forward motion by overcoming the challenges associated with achieving maximum thickness in 3D-printed copper

components and by thoroughly studying their mechanical qualities and exhaustion conduct. [3]

II. PROBLEM STATEMENT

Conventional manufacturing procedures for copper-based components commonly encounter problems when trying to produce complicated geometries and maximize thermal and electrical conductivities. This study plans to show copper-based products created by a two-stage, pressure-based sintering technique and 3D stereolithography (SLA) printing technology. In order to promote 3D SLA printing technology and satisfy the demands of businesses that want copper components of high quality with increased thermal and electrical conductivities, the research intends to improve the density, mechanical characteristics, and fatigue behavior of these components.

III. LITERATURE REVIEW

SLA is one of the various copper 3D printing techniques that uses a laser to selectively cure a liquid resin that contains metal powder and resin. The cured components are then unbound and sintered to produce dense metal sections with fewer flaws. To optimize the printing, de-binding, and sintering parameters for SLA of copper and copper composites, several studies have been carried out in the past. The effects and content on the density, porosity, shrinkage, microstructure, and thermal conductivity of the sintered parts, for instance, have been described by [4] Lanzl et al. To comprehend the relationship between the morphology and properties of sintered copper films made by SLA, [5] Kulkarni et al. adopted a multi-method characterization technique. They investigated how various sintering parameters affected grain size, density, porosity, morphology, thermal conductivity, electrical conductivity, surface roughness, and hardness. They discovered that sintering materials at higher temperatures over longer periods of time produced materials with greater density, porosity, grain size, smoothness, hardness, and electrical and thermal conductivities.

In one of [6] Lee's publications, free-from pure copper structures are printed using stereolithography to achieve the appropriate level of electrical conductivity. Low viscosity acrylate-based resin formulation and a resin-recoating mechanism were both developed in conjunction with metal powder suspension. Cu powder and resin concentrations are specified in detail. The suspension was successfully used to create microstructures. The adopted temperature profile and sintering conditions are very optimized and are easily applicable to our job. SLA was used by Robinson et al. to print various compositions of copper-silver alloys and characterize their mechanical, electrical, and thermal characteristics both before and after sintering. They discovered that the density, porosity, shrinkage, and mechanical characteristics of the pieces were significantly impacted by the sintering temperature and duration. [7] Additionally, they assessed the sintered components' electrical and thermal conductivities and contrasted them with those of bulk copper and silver. They claimed that while preserving high electrical and thermal conductivities, the copper- silver alloys had better mechanical qualities than pure copper and silver. These studies show the promise of SLA in making high-quality, enhanced-property copper and copper alloy parts for a range of applications.

IV. METHODOLOGY

A. Characterization of resin

The resin used in our work is Anycubic colorless resin from SHENZHEN ANYCUBIC TECHNOLOGY CO., LTD [8]. Exact composition of the resin is mentioned in “SAFETY DATA SHEET FOR CHEMICAL PRODUCTS”. by New best Testing Service. Main components of the resin are mentioned in the table :

Substance	**% Composition**
Polyurethane acrylate	30-60
Acrylate Monomer	10-40
Photo initiator	2-5

Table 1 Composition of Resin

Physical and chemical properties of the pure resin are mentioned in table:

Property name	**Value**
Density (g/cm^3)	0.8-0.9
Viscosity	150-250 mPa-s
Boiling point (^OC)	238
Odour	Odourless

Table 2 Properties of Resin

In order to determine the exact thermal behavior of the resin, differential scanning calorimetry is performed.

DSC profile of Anycubic resin shows onset glass transition at 294.18 ^{O}C with midpoint Tg of 307.24 ^{O}C.

After 320^{O}C, the resin completely degrades and no response is observed after this temperature. This point is particularly important in determining the debinding temperature for sintering of the samples.

B. Characterization of Cu powder

Cu powder used in this work is 99.8 % pure and has the following composition as shown in table:

Substance	% Composition
Iron (Fe)	0.015
Lead (Pb)	0.03
Water (H2O)	0.05
Arsenic (As)	0.005
Nickel (Ni)	0.003
Tin (Sn)	0.004
Zinc (Zn)	0.004

Table 3 Composition of Cu Powder

Particle size distribution has been estimated through SEM.

C. Premix formation:

Premix here represents the mixture of resin and Cu powder. The premix is formed by mixing a certain %weight of Cu powder into the resin. Since the masses involved here are quite small therefore a minute scale mass balance is used as shown in fig.

Figure 1 Premix Formation

D. Samples preparation:

Initially the samples preparation is carried out using the following scheme as shown in table. A premix sample in each composition below is prepared.

Sample ID	Cu %	Resin %
Sample 1	50	50
Sample 2	55	45
Sample 3	60	40
Sample 4	65	35
Sample 5	70	30

Table 4 Sample Configuration

The reason to follow this variation is to check the optimized composition of both components that show maximum density, minimum viscosity, suitable curing time etc. The configuration which was then selected at the end was 70% Cu and 30% Resin. Because at this proportion the achieving density was maximum. After that, we have mixed a diluting agent Ethylene Glycol to reduce the viscosity of the premix and make it equal to viscosity of the neat resin. So, that the 3D printing of the copper powder be easily possible.

Figure 2 Rheology

Figure 3 Mixture Preparation

Figure 4 Sample Preparation

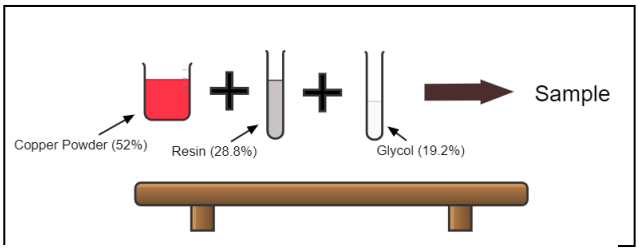

Figure 5 Composition of Mixture

E. Sintering of Sample

In order to obtain good quality samples and keeping in view the oxidizing nature of Cu when exposed to high temperatures, sintering profile used is shown in figure. Our resin was capable of degrading beyond 320^{O}C and therefore any temperature above this value will de-bind the resin from samples. Therefore, the samples are first at around 900^{O}C for 2 hours for removal of resin. In between, temperatures are gradually increased at 10^{O}C/min to avoid Cu oxidation as much as possible [9].

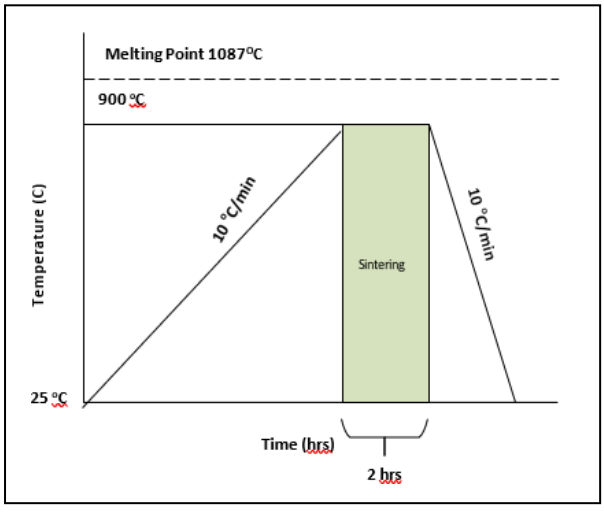

Figure 6 Sintering Profile

F.

F. SEM (Scanning Electron Microscope)

After the sintering process, each sample underwent scanning electron microscopy (SEM) analysis. SEM analysis was conducted to examine various aspects of the samples, including the presence of carbon residues resulting from the burning of the resin, the extent of oxidation in the copper material, the size of porosities within the sample, and the particle size of the copper powder used. [10] SEM analysis provides valuable information about the microstructural characteristics and quality of the sintered copper samples, aiding in the assessment and optimization of the fabrication process.

G. Mechanical Properties Study:

We have used different standardized testing methods to evaluate the mechanical properties of the sintered copper printed products, like Vickers or Rockwell hardness tests are performed to assess the material's resistance to indentation. Also, tensile testing is conducted to determine the strength and elongation properties of the sintered copper components. We have also examined the fatigue behavior of the sintered copper printed products to assess their long-term durability and performance under cyclic loading conditions. Fatigue crack growth analysis is conducted to determine the crack propagation characteristics and fatigue life of the components.

V. DATA ANALYSIS

The data obtained we have obtained from the mechanical properties study, fatigue analysis, and characterization techniques were analyzed statistically using appropriate methods. And a comparative analysis is performed to assess the performance of the sintered copper printed products in comparison to traditionally manufactured counterparts.

VI. RESULTS & DISCUSSION

A. Rheology

For the SLA 3D printing of copper, we have made the viscosity of copper-resin mixture nearly equal to that of neat resin. For this purpose, we have added Ethylene glycol, as a diluting agent. The viscosities of both samples were examined by using rheology. The results of rheometer for neat resin, Cu-Resin (70%- 30%) mixture is shown in figure 7, 9 & 11. Also, we have drawn graphs for each between Viscosity/Speed shown in fig 8, 10 & 11.

	A	B	C	D	E
1		VISCOSITY REPORT		Brookfield Ametek	
2	Step	Viscosity	Torque	Speed	Temperature
3	(#)	(cP)	(%)	(RPM)	(°C)
4	1	120	1	0.5	---
5	2	96	1.6	1	---
6	3	103.2	4.3	2.5	---
7	4	103.2	8.6	5	---
8	5	102	17	10	---
9	6	101.7	33.9	20	---
10	7	102	51	30	---
11	8	103.4	86.2	50	---
12	9	102.6	51.3	30	---
13	10	103.2	34.4	20	---
14	11	103.8	17.3	10	---
15	12	104.4	8.7	5	---
16	13	103.2	4.3	2.5	---
17	14	96	1.6	1	---
18	15	108	0.9	0.5	---
19	16	0	0	0	---
20					

Figure 7 Results of Pure Resin

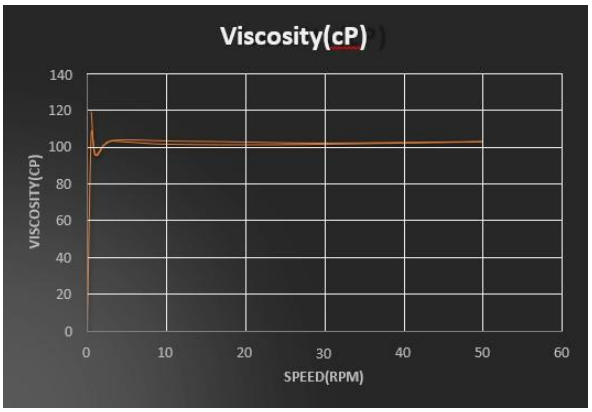

Figure 8 Graph of Pure Resin

A	B	C	D	E
	VISCOSITY REPORT		Brookfield Ametek	
Steps:	Viscosity	Torque	Speed	Temperature
()	(cP)	(%)	(RPM)	(°C)
Step	Viscosity	Torque	Speed	Temperature
(#)	(cP)	(%)	(RPM)	(°C)
1	192	1.6	0.5	---
2	210	3.5	1	---
3	218.4	9.1	2.5	---
4	234	19.5	5	---
5	251.4	41.9	10	---
6	266.7	88.9	20	---
7	270.8	90.1	30	---
8	280	94.1	50	---
9	271.8	89.1	30	---
10	249.6	83.2	20	---
11	249.6	41.6	10	---
12	246	20.5	5	---
13	240	10	2.5	---
14	258	4.3	1	---
15	240	2	0.5	---
16	0	0.3	0	---

Figure 9 Figure 9 Results of Copper + Resin

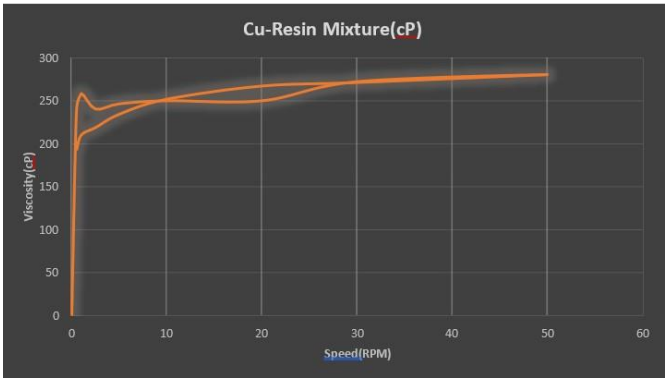

Figure 10 Graph of Copper + Resin

Step	a	Torque	Speed	Temperature	Mean Cp
(#)	(cP)	(%)	(RPM)	(°C)	
1	127	1.4	0.5	---	123.5
2	106	1.9	1	---	101
3	114.8	4.8	2.5	---	109
4	115.2	8.9	5	---	109.2
5	112	117.8	10	---	107
6	111.2	34.1	20	---	106.45
7	114.7	56.3	30	---	108.35
8	115.4	89.1	50	---	109.4
9	111.8	57.2	30	---	107.2
10	114.8	34.5	20	---	109
11	114.2	117.1	10	---	109
12	116.4	9.1	5	---	110.4
13	112.8	4.7	2.5	---	108
14	105.1	2	1	---	100.55
15	119.1	1.7	0.5	---	113.55
16	0	4.6	0	---	0

Figure 11 Results of Mixture + Glycol

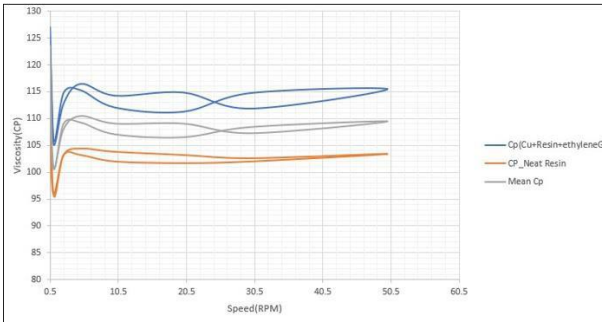

Figure 12 Graph of Mixture + Glycol

B. Sintering Results

Here below are the results for sintering which was done to increase the density of the samples. As initially we have prepared five samples with copper percentages varies as 50%, 55%, 60%, 65%, 70%. We have done sintering for all the five samples. The results for the double phase sintering are as below:

Sr No	Sample ID	Initial volume (cm^3)	Volume after sintering (cm^3)	% Shrinkage	Mass (gms)	Density (g/cm^3)	% Density w.r.t Cu
1.	**Copper 50%**	3.2	1.3	59%	4.2311	3.25469	36%
2.	**Copper 55%**	3.5	1.2	66%	4.7479	3.95658	44%
3.	**Copper 60%**	3.1	1.3	58%	5.5901	4.30008	48%
4.	**Copper 65%**	3.4	1.2	65%	5.6493	4.70775	53%
5.	**Copper 70%**	3.2	1.1	66%	5.7371	5.21555	58%

Table 5 %Shrinkage & Relative Density

C. SEM Results

Below are the SEM results for our product we have made using 3D printing SLA after applying double phase sintering to reduce voids and increasing relative density, have ethylene glycol added to it of configuration of 19.2% of the total mixture.

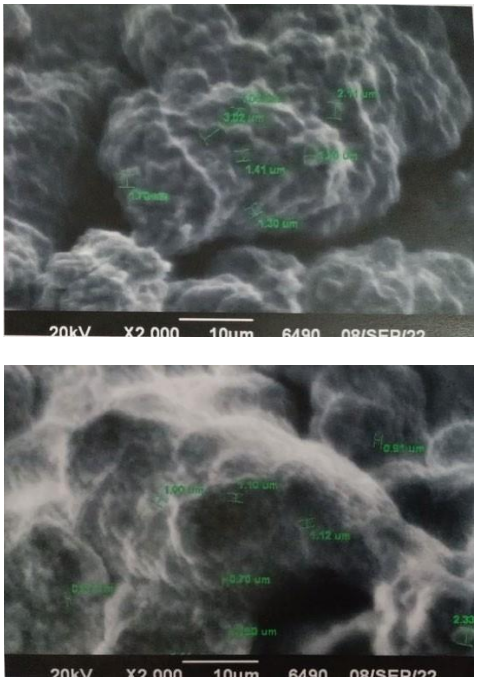

Figure 13 SEM Results of 3D printed Parts

The calculated average grain size for the 3D printed sample was 7.64nm as measured though ImageJ software by analyzing the SEM results of our 3D printed sample (Fig).

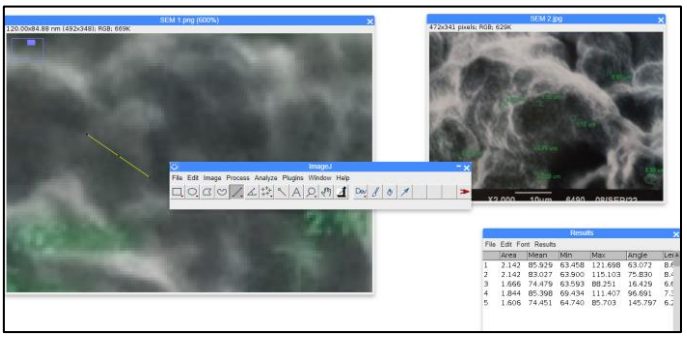

Figure 14 Grain Size Calculation

VII. CONCLUSION

In conclusion, this research project has successfully investigated how complex copper-based components can be produced using 3D stereolithography (SLA) printing technology. The main goal was to improve the 3D metal printing of pure copper and obtain maximum density through a two-phase pressure-based sintering method by utilizing the excellent thermal and electrical conductivities of copper. The presence of voids within the printed copper components was decreased by optimizing the sintering conditions and using regulated pressure, improving material consolidation and structural integrity [11].

This work has offered insightful information through a thorough evaluation of the mechanical characteristics and fatigue behavior of the sintered copper printed goods. Key metrics like hardness, tensile strength, and ductility were examined using standardized testing procedures, and the results were compared to conventionally produced materials. To assess the fatigue performance of the sintered copper components, fatigue crack growth analyses and cyclic loading experiments were also carried out. X-ray diffraction (XRD) and scanning electron microscopy (SEM) are two microstructural analysis methods that made it possible to look at grain size, distribution, and microstructure.

The results of this study have important ramifications for the development of 3D SLA printing technology, especially in the context of copper-based components. This study brings up new options in industries including electronics, telecommunications, and energy systems by effectively producing complicated copper parts with improved mechanical characteristics and dependable fatigue performance. It holds promise for better performance and efficiency in a variety of devices and systems to be able to manufacture high-quality copper components with superior thermal and electrical conductivities.

In general, this research initiative has helped advance

additive manufacturing technologies, particularly in the area of SLA metal printing. For further improvements in the fabrication of copper-based components, the optimization of sintering techniques and the thorough research of mechanical characteristics and fatigue behavior offer invaluable insights. This study has the potential to spur innovation and development in the realm of manufacturing technology by bridging the gap between conventional manufacturing procedures and additive manufacturing.

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